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MEASURING REFRACTIVE INDEX USING THE FOCAL DISPLACEMENT METHOD (POSTPRINT)

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Measuring refractive index using the focal displacement method

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A simple technique is introduced for measuring the refractive index of plane-parallel samples having thickness of the order of a millimeter. The refractive index values are reported for six bulk semiconductors, each index measured at two infrared wavelengths using this method. The values are found to be within a few percent of those in literature for four semiconductors. The other two semiconductors were newly grown ternary alloys (CdMgTe and CdMnTe), for which the refractive index values have not been reported previously at the wavelengths studied here. © 2014 Optical Society of America

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1. Introduction

The minimum deviation method [1,2] is a standard technique for measuring the refractive index of optical materials with high accuracy. However, many materials, especially those newly fabricated in limited size, are often not available in a prism shape and in sufficiently large dimensions to use this method. Techniques utilizing the Lau effect [3] or employing Michelson or Fabry–Perot interferometry [4–6] can be used for refractive index measurement of plane-parallel samples, but the experimental setups for such interferometric methods are somewhat complicated.

For some applications, such as when a material is used as a window, it is unnecessary to know the refractive index value to a high degree of precision. In this paper we describe a technique that is simple in concept and provides the refractive index values within a few percent accuracy without the need to construct a prism from the material. The feasibility

of the technique is demonstrated and it is used to measure the refractive index values of recently fabricated ternary alloy semiconductors CdMgTe and CdMnTe.

2. Principle

Suppose a Gaussian beam in the air is incident upon a thin lens with a focal length f , and a transparent plane-parallel sample with refractive index n and thickness d is inserted normally in the beam at a distance L from the lens, with $L < f$ as shown in Fig. 1. Under paraxial conditions, propagation of light through the sample is equivalent to propagating through a distance d/n in air [7], assuming the refractive index of air to be unity. Thus, when the sample is inserted, the position of the beam focus shifts farther away from the lens by a distance

$$\Delta z = d - \frac{d}{n} = d(1 - n^{-1}). \quad (1)$$

By experimentally measuring the sample thickness d and the focal displacement Δz , the refractive index can be obtained using Eq. (1).

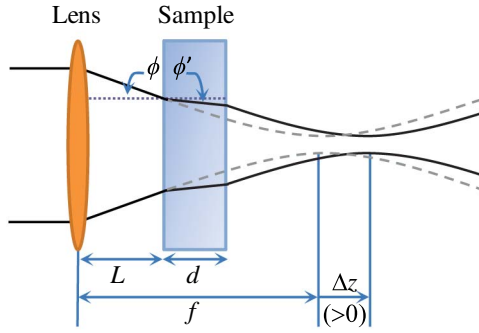


Fig. 1. Sample-induced focal shift.

For strongly convergent beams for which the paraxial condition is violated, marginal rays undergo a different axial shift than paraxial rays. An incident ray making an angle ϕ with respect to the sample normal will be refracted to an angle ϕ' in the sample, and the exiting ray is shifted in z a distance [8]

$$\Delta z = d \left(1 - n^{-1} \left(\frac{\cos \phi}{\cos \phi'} \right) \right). \quad (2)$$

The resulting focal shift must be calculated numerically for such cases. However, for propagation of light between planes in the focal region, the paraxial approximation can remain valid even for strongly focused beams [9] and for a sample placed near the focus, Eq. (1) remains a valid expression for the focal displacement, as the paraxial limit of Eq. (2). Physical optics modeling using the commercial software Zemax [10] confirms that in the paraxial limit, the focal shifts observed with the sample position fixed immediately after the lens match those observed when the sample is translated with the detector through focus.

3. Experiment

Sample thickness (d) was measured for each sample using a Beta LaserMike optical micrometer (Model 60-05-01). Focal displacement (Δz) in the presence and the absence of the sample was determined by the axial translation of a detector with a pinhole, as described below in detail.

The laser beams used had wavelengths of 4.8, 4.64, and 3.39 μm and were obtained from a pulsed frequency-doubled Laser Science TEA CO_2 laser, a frequency-doubled quasi-continuous wave Coherent DEOS Mid-IR-2 CO_2 laser, and a continuous wave Research Electro-Optics He:Ne laser, respectively. The frequency-doubled Laser Science beam had a pulse duration of about 80 ns and a pulse repetition rate of 5 Hz, and the Coherent DEOS and Research Electro-Optics laser beams were chopped at a frequency of 50 Hz. In each case, the laser beam was spatially filtered (producing a Gaussian-like beam), collimated, attenuated if necessary, and steered by gold mirrors to illuminate a 1" diameter anti-reflection coated aspherical ZnSe lens (ISP Optics) having a 1" focal length (see Fig. 2). The Rayleigh

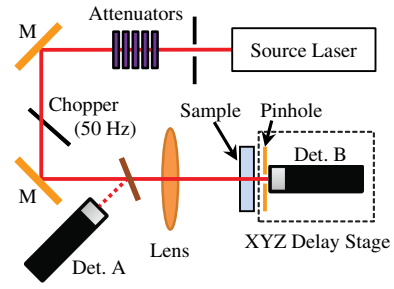


Fig. 2. Experimental setup.

ranges in air were 135, 388, and 69 μm at the wavelengths of 4.8, 4.64, and 3.39 μm , respectively. The laser beams were aligned along the z -axis of the translation stage to minimize detector walk-off.

When the paraxial condition is met, Eq. (1) is valid for any sample position between the lens and its focus. However, due to the size of the sample mount and the short focal length of the lens, placing the sample at a fixed position after the lens limited the distance through which the detector could be translated. To increase this distance, the sample was mounted on the pinhole-detector combination, and the three elements were moved together. The laser power was maintained at values low enough to ensure that no nonlinear effects occurred during these experiments.

The focal position was determined by measuring the beam transmission through a pinhole, probed by a detector "B". To account for any fluctuations in the incident laser power, a beam-splitter was used to reflect part of the beam onto a detector "A". The recorded signal was the ratio of the two detector measurements, B/A .

For detector B, a PbSe photodiode was used (ThorLabs PDA 20H). The photodiode signal was measured by a digital lock-in amplifier (Stanford Research Systems SR830) in the case of the 4.64 and 3.39 μm laser beams, and by a digital oscilloscope (Tektronix TDS 5104) in the case of the pulsed 4.8 μm beam. For the 4.8 and 4.64 μm lasers, detector A was a photodiode (Boston Electronics PVM-10.6) connected to the Tektronix TDS 5104 oscilloscope; for the 3.39 μm laser, stability was such that no

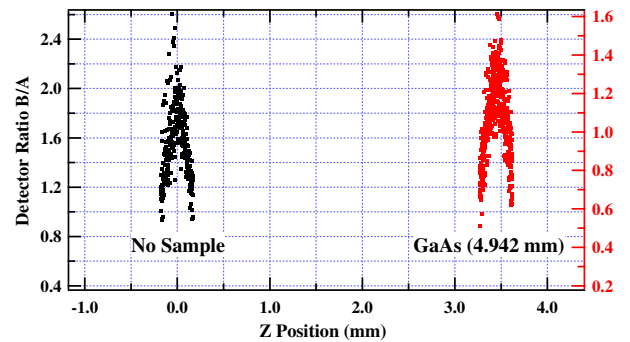


Fig. 3. Displacement scan at 4.64 μm with and without GaAs sample. The right axis gives the scale (a.u.) when the sample is present (Fresnel losses).

Table 1. Measured Refractive Indices Using the Focal Displacement Method and Comparison to Published Result

| Sample | Thickness (mm) | λ (μm) | Δz (mm) | n_{exp} | $\delta n_{\text{exp}}/n_{\text{exp}}$ | n_{pub} | Error |
|--------|----------------|-----------------------------|-----------------|------------------|--|------------------|-------|
| Ge | 1.235 | 3.39 | 0.93 | 4.10 | 3.9% | 4.034 [12] | 1.5% |
| | | 4.8 | 0.91 | 3.83 | 3.9% | 4.018 [12] | 4.7% |
| CdTe | 2.251 | 3.39 | 1.42 | 2.69 | 1.2% | 2.695 [13] | 0.1% |
| | | 4.8 | 1.41 | 2.68 | 1.2% | 2.683 [13] | 0.2% |
| InAs | 1.006 | 4.64 | 0.71 | 3.40 | 7.3% | 3.475 [14] | 2.0% |
| GaAs | 4.942 | 4.64 | 3.43 | 3.28 | 2.2% | 3.298 [11] | 0.6% |

reference detector was required (i.e., the “A” signal was assumed constant).

The PbSe photodiode (B) was placed behind a 5 μm diameter pinhole and mounted on an xyz stage. The pinhole diameter was smaller than the beam waist diameters (FW $e^{-2}M$) of approximately 25, 53, and 20 μm at the wavelengths of 4.8, 4.64, and 3.39 μm , respectively. With the pinhole-detector combination near focus, the x - and y -positions were adjusted to maximize the signal B/A. The signal was then recorded at each z -position, and the position of the peak indicated the focal position. The measurement was repeated with the sample mounted on the front of the pinhole. The difference in focal positions, with and without the sample, is the focal displacement Δz . From the values of Δz and d for each sample, the value of n was calculated using Eq. (1).

4. Results and Discussion

Measurements were conducted first for four samples of materials with refractive index values known from the literature. These were Ge, CdTe, InAs, and GaAs. This technique was then used to measure the index values of two newly grown ternary semiconductors, CdMgTe and CdMnTe, for which there are no bulk index values available. The laboratory temperature during these measurements was 19°C.

Typical data is shown in Fig. 3 where the relative energy transmitted through the pinhole is plotted as a function of the axial distance, with and without a GaAs sample (II-VI, Inc.) mounted in front of the pinhole. The sample was 4.942 ± 0.004 mm thick and the sample-induced focal displacement was 3.43 mm—corresponding to a refractive index of 3.28 ± 0.07 [Eq. (1)]. Compared with the accepted

value of 3.298 [11] (at 19°C), this measurement has an error of 0.6%.

Table 1 lists the results of measurements on GaAs and three other samples along with previously published refractive index values. Both the measurement uncertainties ($\delta n/n$) [15] and the errors (with respect to the published values) are within a few percent for all samples, indicating the validity of the method. The uncertainties were largest for Ge and InAs, arising from a wedge of 2.5 mrad for the Ge sample and from the relatively small sample thickness d for InAs (since the uncertainty depends on the ratio $\Delta z/d$). Measurement uncertainties can be reduced by using thicker samples with better surface parallelism.

For non-wedged samples, the standard deviation of the thickness measurement was ~ 4 μm ; for wedged samples, the uncertainties were greater (~ 10 μm for the Ge sample studied). The thickness uncertainty can be reduced by improving the parallelism of the sample faces. The uncertainty in Δz arises from the error in locating the focal position (which is a function of the Rayleigh range of the beam) and from the positioning errors due to the actuator and translation stage positioning.

Positioning uncertainty was minimized by allowing for actuator travel only in one direction. For the Newport LTA-HL precision motorized actuators used, the manufacturer’s guaranteed actuator unidirectional repeatability was ± 0.25 μm (3 sigma), or a standard deviation of 0.1 μm . The Newport 426 translation stage used has a pitch error less than 150 μrad . Since the pinhole was located 121 mm above the translation stage, the pitch retards or advances the pinhole less than 18 μm . Although this is an upper limit, it is rather large (a few percent of

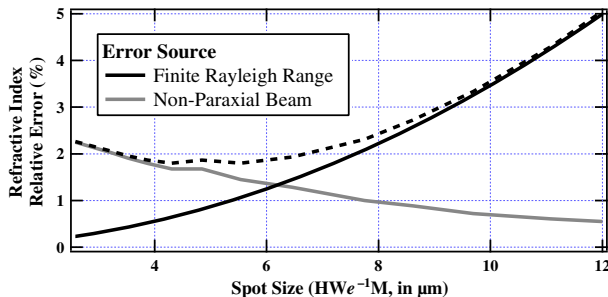


Fig. 4. Calculated relative errors owing to finite Rayleigh range (black) and the use of the paraxial approximation (gray). The dashed curve represents their sum in quadrature. Calculation is for a $d = 3$ mm GaAs sample at $\lambda = 3.39$ μm .

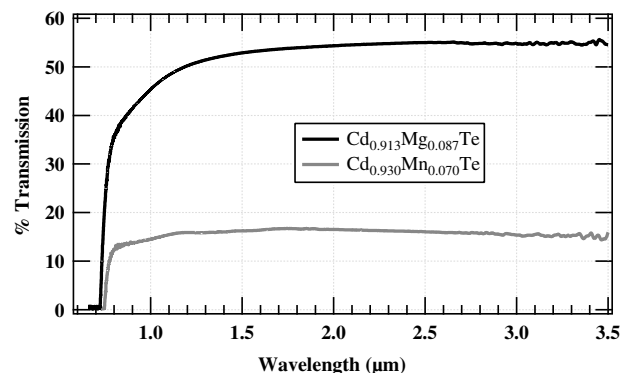


Fig. 5. Measured spectra for CdMgTe (black) and CdMnTe (gray).

Table 2. Refractive Index of CdMgTe and CdMnTe at 292 K as Found Using the Focal Displacement Method

| Sample | Thickness (mm) | Wedge (mrad) | λ (μm) | Δz (mm) | n_{exp} | $\delta n_{\text{exp}}/n_{\text{exp}}$ |
|--|----------------|--------------|-----------------------------|-----------------|------------------|--|
| Cd _{0.913} Mg _{0.087} Te | 2.980 | 0.4 | 3.39 | 1.86 | 2.65 ± 0.02 | 0.8% |
| | | | 4.8 | 1.88 | 2.63 ± 0.02 | 0.9% |
| Cd _{0.930} Mn _{0.070} Te | 2.460 | 2.1 | 3.39 | 1.51 | 2.59 ± 0.03 | 1.1% |
| | | | 4.8 | 1.53 | 2.65 ± 0.03 | 1.2% |

the typical ~ 1 mm displacement distance) and may introduce systematic rather than random error. In spite of these large uncertainties shown in the column labeled $\delta n_{\text{exp}}/n_{\text{exp}}$ in Table 1, the results presented here match the literature values to within 2% in all but one case. The exception was the measurement at 4.8 μm of a Ge sample with known wedge (2.5 mrad) and consequently larger thickness uncertainty.

To choose an appropriate spot size that minimizes the uncertainty in Δz , we note that while the focal positions can be located more precisely by minimizing the Rayleigh range, i.e., by reducing the focal spot size, for this simple technique to be applicable the spot size should not be reduced below the values for which Eq. (1) remains valid. For a sample of given thickness and for light of a given wavelength, the optimum Rayleigh range and spot size can be determined numerically. For example, for the case of GaAs at 3.39 μm with a hypothetical sample thickness of 3 mm, the relative error $\delta n/n$ with respect to the value $n = 3.308$ [11] (19°C) was calculated for a range of beam radii at an $f = 1''$ lens using a physical optics model in Zemax (Fig. 4). Also plotted in Fig. 4 is the calculated refractive index uncertainty, neglecting any thickness uncertainty, introduced by the finite Rayleigh range alone. (It was found empirically that the uncertainty of the focal position was approximately 12% of the Rayleigh range.) Although the former represents a systematic and the latter a random error, it is instructive to add the curves in quadrature (dashed curve), from which it is seen that a focal spot size of 5 μm (HWE^{-1}M) is optimal. This corresponds to a Rayleigh range of 47 μm and a 2.7 mm spot size at the lens.

Thus, the errors in the refractive index measurements can be reduced further from the measurements presented here by better sample polishing, faster focusing, lower pinhole placement, use of a translation stage with tighter tolerances, and using thicker samples if available.

The focal displacement method described above was used to measure the refractive index values for two newly grown samples of CdMgTe and CdMnTe. These materials show promise as x-ray and gamma-ray detector materials [16,17] and also in nonlinear optics [18], but they are not easily available commercially. Their refractive indices are therefore not well known, and our review of the literature yielded no room temperature CdMgTe (CdMnTe) refractive index data at wavelengths longer than 1.8 μm [19] (2.5 μm [20]).

The CdMgTe and CdMnTe crystals were obtained from Brimrose Technology Corp. The linear transmission spectra of the two samples are shown in Fig. 5. Electron probe micro analyzer measurements revealed the CdMgTe sample to have a Mg fraction of 8.7% and the CdMnTe sample to have a Mn fraction of 7.0%. X-ray diffraction scans showed each sample to be a single crystal with orientation (110). Their refractive index values measured using the focal displacement method at two different wavelengths are listed in Table 2.

Both samples suffered from a slight wedge, and the uncertainty in the refractive index measurements was around 1%, which is similar to that seen with the other samples. The results show that the refractive index values follow the trends shown in [19] and [20], and their wavelength dependence in the mid-wave infrared spectral region is relatively weak.

5. Conclusion

A simple method to measure the refractive index of plane-parallel materials is described. The method yields results accurate to within a few percent, and some ways to improve its accuracy were identified. The technique was demonstrated for materials with known refractive indices and for two ternary semiconductor compositions for which the refractive indices are not found in the literature. Use of a camera, as suggested by the work of Sun *et al.* [21], to locate the focal position will simplify the technique and possibly increase its accuracy.

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15. From Eq. (1), the refractive index is given by $n = (1 - \Delta z/d)^{-1}$. With the reasonable assumption that the thickness measurement errors are normally distributed and uncorrelated with Δz , the relative uncertainty $\delta n/n$ in the refractive index is

$$\frac{\delta n}{n} = n^{-1} \sqrt{\left(\frac{\partial n}{\partial d} \delta d\right)^2 + \left(\frac{\partial n}{\partial \Delta z} \delta \Delta z\right)^2} = \frac{n}{d} \sqrt{\left(\frac{\Delta z}{d} \delta d\right)^2 + (\delta \Delta z)^2}.$$

The uncertainty $\delta \Delta z$ in the focal shift is given by

$$\delta \Delta z = \sqrt{\delta z_{\text{sample}}^2 + \delta z_{\text{nosample}}^2} \approx \delta z \sqrt{2},$$

where we have explicitly assumed that the focal position measurement error δz is unchanged by the presence of the sample.

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